

THE RESULTS OF THE VACUUM CHARACTERISTICS MEASUREMENTS OF UHV PROTOTYPE FROM THE SINTERED NEG DISKS FOR HYDROGEN AND ITS ISOTOPES

A. Semenov^{1, 2 *}, A. Krasnov^{1, 3}

¹ Budker Institute of Nuclear Physics, Novosibirsk 630090, Russia

² Novosibirsk State Technical University, Novosibirsk 630073, Russia

³ Novosibirsk State University, Novosibirsk 630090, Russia

Abstract — Nowadays it is impossible to imagine an ultra-high vacuum system without the use of vacuum pumps based on non-evaporable getters. This paper describes the construction of non-evaporable getter pump from modern domestic materials. The measured characteristics for hydrogen and its isotopes for the application in synchrotron sources (for example, Synchrotron Research Facility «SKIF») and in fusion research will be presented as well. For example, in the vacuum system of neutral beam injectors (hydrogen and deuterium pumping speeds are equal millions of liter per second by pressure of 10^{-5} Torr). Effective pumping speed of prototype of the order of 1300 l/s for hydrogen and 700 l/s for deuterium were achieved. These values are in good agreement with understanding of the processes in the behavior of the non-evaporable getter pump during evacuation of hydrogen and deuterium. The reasons why the effective pumping speed for deuterium is less by two times rather than the square root from two is discussed.

Keywords—ultra-high vacuum, synchrotron source, deuterium, pumping speed, non-evaporable getter, plasma.

INTRODUCTION

The ambitious goal for researches for the last decades – creating a reactor based on controlled thermonuclear fusion. A large number of research teams have been working on the problem. In a thermonuclear reaction, light atomic nuclei combine to form heavier ones. This process occurs in plasma during its combustion. Thermonuclear fusion happens under very high temperature conditions, since combustion requires heating the plasma to one hundred million Kelvin's degrees. A reactor based on controlled thermonuclear fusion is capable of providing humanity with cheap energy for many years [1].

Physicists consider pure deuterium as one of the possible options as a fuel for controlled thermonuclear fusion - an unlimited resource that literally falls on us from the sky. Each cubic meter of water contains thirty-three grams of deuterium; this source of energy is practically inexhaustible.

Condensation-type cryosorption pumps cooled by liquid helium and cryocoolers based on the Gifford-McMahon cycle, as well as cryopumps with a speed of hundreds of thousands of liters per second for hydrogen, have been used as the main means of pumping hydrogen out of neutral beam injectors at the BINP recently. Neutral-beam injection (NBI) is one method used to heat plasma inside a fusion device consisting in a beam of high-energy neutral particles that can enter the magnetic confinement field. When these neutral particles are ionized by collision with the plasma particles, they are kept in the plasma by the confining magnetic field and can transfer most of their energy by further collisions with the plasma. To date, all NBI systems were based on positive precursor ion beams. The use of cryosorption pumps in neutral injectors is difficult due to the limited space available for placing the pumping system near the beam transport area. It can be economically unprofitable, since it requires constant maintenance of cryogenic temperatures under a significant thermal load. Otherwise, even with slight heating of the cryopane, the hydrogen condensed on it begins to desorb. The fundamental limitation is the location of the beam source and the transport path under high voltage (up to one megavolt), which requires a high-voltage decoupling between the injector cryopumps and the liquid and gaseous helium supply/reception system [2].

* corresponding author's email: a.m.semenov@inp.nsk.su

That is one of the reason to NEG pumps are being promising alternative for cryogenic pumps in application in fusion research. In particular, it requires high hydrogen pumping speed and large sorption capacity for the vacuum system of neutral beam injectors where gas is injected continuous.

The vacuum specialists have a large variety of different means of obtaining vacuum based on getters, there is currently no universal solution. These are lumped pumps (cartridges of non-evaporable getters (NEG) installed through connecting flanges inside the vacuum chamber) and distributed pumps (non-evaporable getters coated to the inner surface walls of vacuum chambers).

To sorb most of the active gas, except inert gases and methane, non-evaporable getters are used as sorbed vacuum pumps in the different forms [3], [4]. NEG alloys were well studied for the possibility of application in ultra-high vacuum, for example, Ti, Zr, V, Hf, rare earths, etc [5]. For the first time, they were used in ultrahigh vacuum system as well as in the Large Electron Positron collider yet in 1970s [6]. And since that time NEG has been an integral part of vacuum technology. In synchrotron light machines non-evaporable getters are used to minimize the gas load [7] as distributed coatings. The high ultimate solubility of oxygen for elements of group VI of the periodic Mendeleev's Table allows for multiple passivation (opening to the atmosphere)/reactivation cycles at the activation temperature without a noticeable decrease in the pumping speed.

Next step in development of non-evaporable getters is a combination the pumps based NEG with sputter ion pumps [8] able to evacuate inert gases and methane for the high vacuum and ultra-high vacuum.

Among active gases, interacting with the getter, hydrogen and its isotopes have a peculiar behavior. They are sorbed in a reversible way. In typical conditions, hydrogen dissociates at the solid surface, and diffuses in the getter material. This provides a stable pumping speed over time as the bulk of the material makes a contribution to the sorption capacity, not only the surface. A limit to the sorption capacity for NEG elements is cracking due to saturation by hydrogen, when subject to cyclic hydrogen loads reaching concentrations above the recommended ones. The behavior under sorption and regeneration conditions allows the NEG application in machines requiring large hydrogen pumping.

In Russia, one of the manufacturers of sintered non-evaporable getters is the Polema company.

This paper describes the development of a NEG prototype pump with a pumping speed of 1300 l/s for hydrogen and 700 l/s for deuterium for its performance to synchrotron light sources and very large pumps such as for neutral beam injectors. Also, here is a reason, why the effective pumping speed for deuterium is less by two times rather than the square root from two is discussed.

DESCRIPTION OF NEG PROTOTYPE

«Polema» LLC has produced non-evaporable getters in large quantities for the nuclear power and oil and gas industries [9]. The first vacuum prototype pumps based on Ti–Zr–Al getters manufactured by «Polema» LLC were made from tablets 13 ± 0.5 mm in diameter and 3 ± 0.5 mm thick, with 60% porosity. More detailed results can be found in [10].

The main parameters of the prototype is presented in Table 1. The prototype is made of getter discs. Each disc has an outer diameter of 25 mm and is 1.6 mm thick. The porosity is 30%, and the inner diameter is 8 mm to locate the heater. The chemical composition of the non-evaporable getter is Ti–Zr–Al. The discs are installed in series in columns with gaps of 1.5 mm. There are 31 pcs in each column. The total number of columns is equal of 6 pcs. The height (from the flange to the end face of the getter pump) is no more than 130 mm. The temperature is controlled with a chromel-alumel thermocouple. One protective screen is used outside to reduce the heater's power by half. The getter pump is on flanged connections of the Conflat type. Images of getter pump is shown in Fig. 1.

TABLE 1. MAIN PARAMETERS OF THE NEG PROTOTYPE

Name	Activation	NEG Surface,	NEG	Porosity,	Flange
------	------------	--------------	-----	-----------	--------

	Temperature, °C	cm ²	Weight, g	%	Type
NNG-1,3	650	1600	360	30	DN 100



Fig. 1. Images of getter pump NNG-1, 3.

EXPERIMENTAL SETUP AND MEASURING TECHNIQUE

The activation and gas sorption characteristics of the getter pump were measured on a specialized experimental setup (Fig. 2). The system was preliminarily evacuated by a station with a turbomolecular pump (*TMP1*) and an oil-free membranous pump (*MP*). High-vacuum pumping was achieved by the turbomolecular pump *TMP2* through vacuum all-metal valves *VR2*. The typical pressure level of turbomolecular pump *TMP2* is equal down to $1\text{E-}10$ Torr. An arrow-like type pressure manometer and a Baratron capacitive gauge were used to monitor the pressure of the injected gas.

Gas was let into the system through leak valve *VF* and capillary *C* (the molecular conductivity with respect to hydrogen was $2.8\text{E-}3$ l/s). The injection system was evacuated by the turbomolecular pump *TMP1* and an oil-free membranous pump *MP* through vacuum all-metal valves *VR4* and *VR5*. Emergency electric valve *VR3* was installed to prevent the atmosphere from breaking through from the *TMP2* and *TMP1*. The fore and high vacuums were measured at the pumping station by a Pfeiffer PKR 251 full range pressure sensor consisting of a Pirani sensor and a cold cathode gauge.

The pressure in the system was measured by hot cathode gauge *IG1*. The partial pressures of gases were measured using an *RGA* quadrupole mass spectrometer from SRS (Stanford, United States). Hot cathode gauge *IG2* was needed to control the gas flow pumped out by turbomolecular pump *TPM2*.

Since vacuum system in SRF «SKIF» and in neutral beam injectors will not be baked out, the setup was not baked as well. It was measured the effective pumping speed of turbomolecular pump *TMP2*, the molecular conductivity of the capillary, and the coefficients of sensitivity of the pressure sensors with hot cathodes for each gas (H_2 , CO and D_2). Table 2 gives these values for each gas. All vacuum gauges were calibrated each time after vent of air atmosphere. Valve *VR1* was closed at the start of the experiments. It was then opened, and the injected gas began to flow into the test chamber through the capillary.

The gas flow into the test chamber can be determined as:

$$Q_{total} = C \cdot (P_{bar} - P_{IG1}), \quad (1)$$

where C is a capillary with calibrated conductivity, l/s; P_{bar} and P_{IG1} are the pressures by the capacitive gauge and hot cathode gauge, Torr.

Pumping speed S_{NEG} of the getter during gas injection can be determined as:

$$S_{NEG} = \frac{C \cdot (P_{bar} - P_{IG1})}{K(P_{IG1after} - P_{IG1before})} - S_{TMP2}, \quad (2)$$

where S_{NEG} is the pumping speed of the NEG pump. S_{TMP2} is the pumping speed of the turbomolecular pump: 28 l/s for hydrogen, 8.5 l/s for carbon monoxide and 24 l/s for deuterium. C is the molecular conductivity of the capillary l/s; $P_{IG1before}$ is the pressure of hot cathode gauge $IG1$ before the gas inlet, Torr; $P_{IG1after}$ is the pressure at hot cathode gauge $IG1$ after the gas inlet, Torr; K is the coefficient of sensitivity.

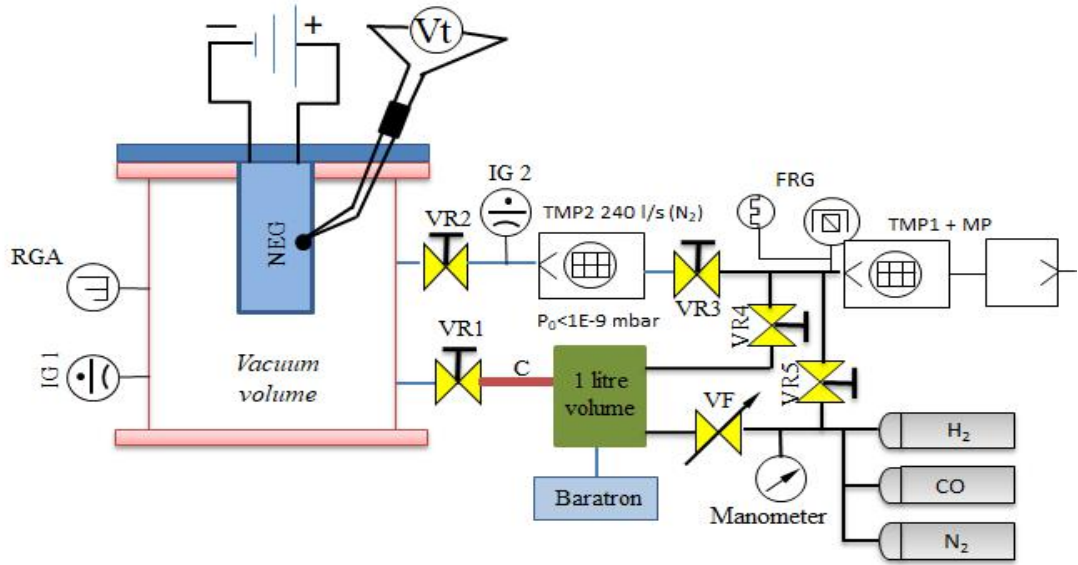


Fig. 2. Simplified scheme of the vacuum setup. RGA is a mass spectrometer; IG1 and IG2 is a hot cathode gauges; FRG is a full range sensor; NEG is the pump being tested; C is a capillary with molecular conductivity; TMP1+MP is a turbomolecular pumping station; TMP2 is a turbomolecular pump; the Baratron is a capacitive gauge; the manometer is a arrow deformation pressure gauge; VF is a slotted leak; VR1, VR2, VR4, and VR5 are all-metal angle valves; and VR3 is the emergency electric valve.

TABLE 2. MOLECULAR CONDUCTIVITY OF THE CAPILLARY, EFFECTIVE PUMPING SPEED OF TURBOMOLECULAR PUMP 2, AND COEFFICIENT OF SENSITIVITY OF HOT CATHODE GAUGES FOR DIFFERENT GASES [11]

Gas	Molecular conductivity C , l/s	Pumping speed of S_{TMP2} , l/s	Coefficient of sensitivity, K_{IG1}	Coefficient of sensitivity, K_{IG2}
H ₂	2,8E-3	28	2,3	2,3
CO	7,6E-4	8,5	1	1
D ₂	2,15E-3	21	3,55	3,55

Sorption capacity was defined as the number of molecules upon whose sorption the pumping speed falls to 10% of the initial level:

$$Doze = \int_0^t (Q_{total} - Q_{TMP2}) \cdot dt, \quad (3)$$

where Q_{total} is the total gas flow, l·Torr/s; Q_{TMP2} is the gas flow pumped out by the turbomolecular pump $TMP2$, l·Torr/s; and t is the period of measuring.

The gas flow pumped out by the turbomolecular pump is determined as:

$$Q_{TMP2} = S_{TMP2} \cdot K \cdot (\Delta P_{IG1} - \Delta P_{IG2}), \quad (4)$$

where

$$\begin{aligned} \Delta P_{IG1} &= P_{IG1after} - P_{IG1before}, \\ \Delta P_{IG2} &= P_{IG2after} - P_{IG2before}, \end{aligned} \quad (5)$$

$P_{IG1before}$ is the pressure of hot cathode gauge $IG1$ before the gas inlet, Torr; $P_{IG1after}$ is the pressure at hot cathode gauge $IG1$ after the gas inlet, Torr; $P_{IG2after}$ is the pressure at hot cathode gauge $IG2$ after the gas inlet, Torr; $P_{IG2before}$ is the pressure of hot cathode gauge $IG2$ before the gas inlet, Torr and K is the coefficient of sensitivity.

RESULTS AND DISCUSSIONS

After pumping down and leakage check at room temperature, each time before measurement the getter was activated for 1 h at the maximum temperature (650°C), and passivated in air for at least 3 h after each measurement. On present day, the quantity of activations and passivations is equal 45 pcs. The test is ongoing.

Measurements of the pumping speed for hydrogen, deuterium and the sorption capacity for carbon monoxide were made for the getter pump at room temperature. The hydrogen and its isotopes sorption capacity was estimated at around several thousand l·Torr, so this sorption capacity is enough after the getter is passivated by oxides. Excessive saturation of the getter with hydrogen also results in destruction and cracking of the getter.

Fig.3 presents the activation temperature versus the heater power [12]. This activation power is given for a vacuum volume with a diameter of 350 mm. It can be expected that with a diameter less than this, the power will also decrease, since heat loss due to radiation will be re-reflected from the walls of the vacuum volume and returned to the getter. The smaller the gap between the NEG pump and the volume wall, the greater the probability of heat return.

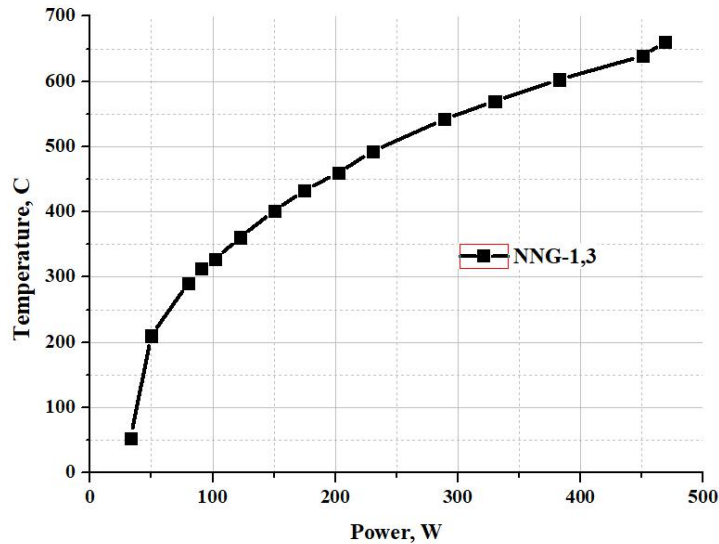


Fig.3. The activation temperature versus power of heater for NEG pump NNG-1,3.

Fig.4 shows the sorption capacity for hydrogen, deuterium and carbon monoxide as a function of the sorbed gas quantity.

NEG pumps are trapping pumps based on the adhesion of gas particles to the adsorption areas of the surface; for a presented adhesion rate, the pumping speed depends on the collision rate, which depends on the arithmetical mean velocity and, therefore, on the square root of the particle mass. In principle, the chemical activity of hydrogen and its isotopes does not depend on the isotope mass. Under such conditions, the pumping speed of deuterium should be 1.41 times lower than one of hydrogen. However, the diffusion coefficient of a dissociated atom from the surface into a bulk material depends on its mass, and this effect can affect the overall pumping speed [13]. According to [14], another explanation may be that even a small content of helium impurities (less than 1%) can lead to a decrease in the pumping speed by a factor of two for deuterium. However, any of these statements requires additional experimental research or the creation of a sound physical model.

The overestimated values of the pumping speed for carbon monoxide are explained by the fact that any "clean" surface has the ability to evacuate gases, although the sorption capacity is usually equal to one monolayer. Therefore, the protective shield and flange of the NEG pump are subjected to significant heating during the activation, after what it is able to absorption. A sharp decline indicates saturation of the surface. The pumping speed of the carbon monoxide pump is equal 850 l/s approximately.

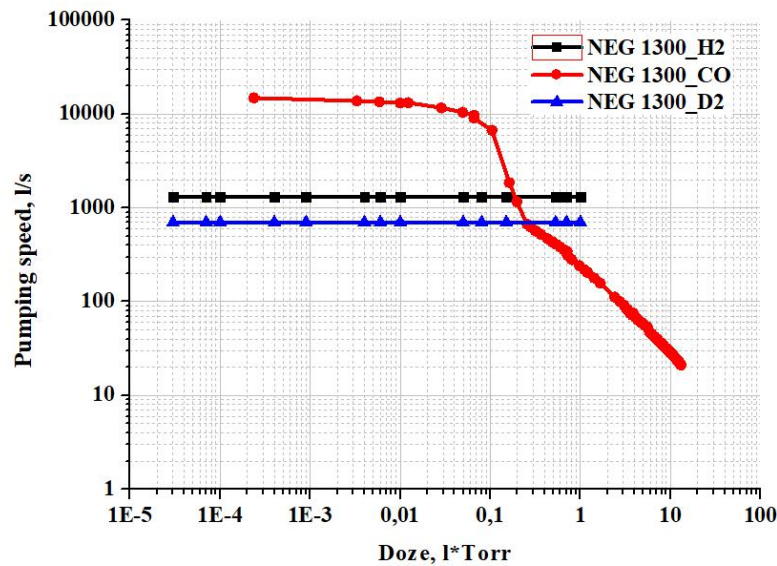


Fig.4. The sorption capacity for hydrogen, deuterium and carbon monoxide as a function of the sorbed gas quantity.

Since the surfaces of getter are easily passivated by some monolayers of oxides and carbides during the pumping down and then chemically bound atoms of oxygen, carbon, and nitrogen diffuse from the surface into the volume of the getter during the activation. In the future, it is necessary to understand how many activation cycles and openings to air to atmospheric pressure are possible without deterioration of the gettering and activation properties of the pump.

A MOCKUP PUMP SUGGESTION FOR NEUTRAL BEAM INJECTORS

As it was said earlier, the construction and investigation of the mockup pump with required large pumping speed (up to 10^3 m³/s) is an important step in the technological creation, development and research of large-scale NEG pumps for the application to neutral beam injectors. As discussed earlier, pumping speed measurement and sorption capacity for hydrogen and deuterium were investigated experimentally on the vacuum pump prototype. Experimental results had also embraced the reliability of thermal heaters and quantity of passivations and activation.

The next step is the creation of mockup pump consisting of more than twenty NEG cartridges with pumping speed of 1300 liter per second. That task rise a question how to locate of heaters, i.e. to place the heater within each cartridge or heaters between the cartridges.

It is expected that the total pumping speed of the mockup pump will be less than the sum of all cartridges separately. The explanation lies in the limited conductivity and/or incomplete activation of some cartridges.

CONCLUSIONS

Prototype of vacuum pumps based on non-evaporable getters with pumping speeds of 1300 l/s for hydrogen and 700 l/s for deuterium was tested. This pump corresponds to foreign counterparts in terms of such characteristics as temperature of activation, pumping speed, and sorption capacity. Based on the obtained results, the production of getter pumps was established in Russia for the first time, which are already being installed in the vacuum chambers of the SKIF storage ring. The tested pumps can be used in both accelerator technology and plasma installations.

REFERENCES

- [1] Yu I Belchenko, V I Davydenko, P P Deichuli, et al. Studies on ion and neutral beam physics and technology in the Institute of Nuclear Physics SB RAS. *Physics – Uspekhi* 61 (6) 531 – 581 (2018).
- [2] Deichuli P., Davydenko V., Belov V., Dranichnikov A. N., *Ivanov A. A.* Commissioning of heating neutral beams for COMPASS-D tokamak. *Rev. Sci. Instrum.*, 2012. Vol. 83. No. 2. Pt. 2. P. 02B114
- [3] P. Manini, A. Conte, L. Viale, A. Bonucci, L. Caruso, A novel route to compact, high performance pumping in UHV–XHV vacuum systems, *Vacuum* 94 (2013) 26–29.
- [4] C. Benvenuti, P. Chiggiato, P. Costa Pinto, A. Prodromides, V. Ruzinov, Influence of the substrate coating temperature on the vacuum properties of Ti–Zr–V nonevaporable getter films, *Vacuum* 71 (2003) 307–315.
- [5] C. Benvenuti, P. Chiggiato, F. Cicoira, and Y.L' Aminot. Non-evaporable getter films ultrahigh vacuum applications, *J. Vac Sci. Technol. A* 16 (1), 148 (1998).
- [6] C. Benvenuti. A new pumping approach for the large electron positron collider (LEP). *Nuclear Instruments and Methods in Physics Research*, vol. 205, issue 3, pp. 391-401, 1983.
- [7] A.G. Mathewson, Ultra high vacuum technology for synchrotron light sources, *Synchrotron Radiation News*, Volume 3, 1990 – Issue 1.
- [8] C.D. Park, S.M. Chung, P. Manini, Combination of compact nonevaporable getter and small ion pumps for ultrahigh vacuum systems, *J. Vac. Sci. Technol. A* 29 (2011), 011012.
- [9] Official website of company «Polema» [Online]. Available: <http://www.polema.net/oblasti-primenenija> <http://www.polema.net/oblasti-primenenija.html>
- [10] V. Anashin, A. Krasnov, A. Semenov, “A study of the gettering characteristics of getter pumps for different gases”, *Instruments and Experimental Techniques*, 2020, Vol. 63, No. 6, pp. 893–897.
- [11] A. Krasnov, A. Semenov, “Lumped Ultra-High Vacuum Pumps Based on Non-Evaporable Getters”, *Bulletin of the Russian Academy of Sciences: Physics*, 2023, Vol. 87, No. 5, pp. 568–572.
- [12] A. Semenov, A. Krasnov, D. Idashin. The Preliminary Results of the Pumping Speed Measurements of UHV Pump Based on the NEG for Hydrogen and its Isotopes. *Paper Presented at the IEEE 25th International Conference of Young Professionals in Electron Devices and Materials (EDM)* (Altai, Russian Federation, 28 June - 02 July 2024). DOI: 10.1109/EDM61683.2024.10615194
- [13] E. Sartori, M. Siragusa, P. Sonato, F. Siviero, M. Mura, E. Maccallini, A. Ferrara P. Manini, S. Hanke, C. Day, Development of non evaporable getter pumps for large hydrogen throughput and capacity in high vacuum regimes, *Vacuum* 214 (2023) 112198,
- [14] M. Singleton, C. Griffith, “Evolution of non-evaporable getter pump for tritium handling in the tokamak fusion test reactor,” Report in Lawrence Livermore Laboratory of University of California (UCRL-52584), 1978.